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(54) **Comestible composition containing stabilized aspartame and process for preparing same.**

(57) L-aspartic acid sweetening agent derivatives are stabilized in comestibles containing $\geq 2\%$ moisture by being formulated with cooked aqueous hydrogenated starch hydrolysate having a moisture content of $10 \pm 6\%$. Optionally, glycerine may also be used.

COMESTIBLE COMPOSITION CONTAINING STABILIZED 0196640
ASPARTAME AND PROCESS FOR PREPARING SAME

The present invention relates to comestibles such as chewing gum containing $\geq 2\%$ moisture and made with L-aspartyl-L-phenylalanine methyl ester (APM or aspartame) in which the aspartame has been significantly stabilized against decomposition, during its shelf life, into decomposition products such as diketopiperazine, by being formulated with aqueous hydrogenated starch hydrolystate that has been cooked to achieve a moisture content of about $10 \pm 6\%$. Glycerine may also be added with the cooked hydrogenated starch hydrolysate.

Aspartame which is used extensively in many types of sugarless foodstuffs, or other comestible products, such as chewing gum, is known to readily decompose in the presence of moisture into decomposition products such as diketopiperazine which causes a significant loss in the sweetness properties of such products during their shelf lives. Many attempts have been made by those producing the various types of products in which aspartame is used in order to provide means for stabilizing the aspartame against such decomposition processes. Such means have included encapsulating the aspartame in various film forming materials (U.S. 3,929,988, U.S. 4,384,004 and International Patent Application WO 84/03201), or by saturating an aqueous dispersing agent used in the product with the aspartame (EPA 102,032).

Each of such various prior art means, however, have certain drawbacks. The encapsulation means requires a separate, time-consuming and relatively expensive procedure for encapsulating the aspartame which amounts to a separate processing operation.

1 Saturating the moisture content of a product with
aspartame requires the use of excess amounts of the aspartame
needed to achieve a certain level of sweetness. Since
aspartame is a relatively expensive material, this proposed
5 solution to the aspartame decomposition procedure is a rather
expensive one. Further, it does not prevent the
decomposition of the aspartame into undesired decomposition
products such as diketopiperazine.

10 It is also desired to use aspartame in products
having relatively high moisture contents in order to avoid
the need for stringent anhydrous operating conditions that
are required when producing chewing gum products having very
low moisture contents, i.e., less than 1 to 2%. See in this
regard European Patent Application 82670 and International
15 Patent Application WO 84-10693.

 Further, various of these prior art chewing gum
products which have such very low moisture contents, i.e., of
about 1 to 2%, also have relatively low equivalent relative
humidity (ERH) values, i.e., of the order to about 10 to 25.
20 During the storage of such products under ambient conditions
of about 40 to 70% relative humidity and about 20 to 30°C,
the (low value) ERH properties of such products will cause
such products to absorb relatively large amounts of water
from the atmosphere. Such increased amounts of water will
25 lead to a more accelerated rate of decomposition of any
unprotected APM therein; and will adversely affect other
qualities of the products.

 Thus, such products having very low ERH values
would require that they be processed and wrapped under
30 special atmospheric conditions of low humidity and controlled
temperature. Such conditions are expensive and difficult to
maintain.

1 Equivalent relative humidity (ERH) or relative
vapor pressure is the humidity at which a foodstuff neither
gains nor loses moisture and the figure is expressed as a
percentage. A discussion of ERH relative to food products
5 and a method for the determination thereof is to be found in
"Chocolate, Cocoa and Confectionery," Science and Technology,
Second Edition, 1980, Bernard W. Minifie, AVI Publishing Co.,
Inc. Westport, Connecticut, U.S.A., Appendix I, pp. 672-677.
The test procedure disclosed in such publication is the one
10 used for testing and evaluating the compositions disclosed
herein.

U.S. 4,248,895 discloses the preparation of a dried
non-hygroscopic free flowing powder that is made by drying a
higher polyalcohol, such as hydrogenated starch hydrolysate,
15 with a concentrated protein extract. The dried material has
a moisture content of about 2-6% and may be used as a
sweetener in confections such as chewing gum to provide
products having prolonged shelf life in terms of retained
flexibility and softeners. Other non-sugar sweeteners may be
20 used with the dried, powdered, higher alcohol sweetener such
as L-aspartyl-L-phenylalanine. The dried hydrogenated starch
hydrolysate may also be used in combination with hydrogenated
starch hydrolysate syrup having a moisture content of 15 to
40%.

25 U.S. 4,382,962 discloses the preparation of
sugarless chewing gum made with a specific hydrogenated
starch hydrolysate composition that is designed to replace
all or a portion of the mannitol previously used with xylitol
and/or sorbitol in such products. Cyclamates and the salts
30 of saccharin may be also used in such products when the
product contains a liquid filler (column 4, lines 20-45).

1 Therefore, it has not been readily possible,
heretofore, to economically provide a means for stabilizing
aspartame in chewing gum products having a relatively high
moisture content so as to provide for more optimum
5 stabilization effects.

 The present invention relates to a comestible
composition sweetened at least partially with at least one
L-aspartic acid sweetening agent derivative freely dispersed
therein and comprising stabilizing amounts of a stabilizer
10 adapted, upon the normal commercial storage of said
composition for at least twelve months, to retard or prevent
the decomposition of said L-aspartic acid derivative into
decomposition products which are devoid of sweetening
characteristics to such extent as to allow for the recovery
15 of at least 70% of the formulated amount of said L-aspartic
acid derivative after said storage, said stabilizer
comprising cooked hydrogenated starch hydrolysate, and said
composition comprising moisture in such amounts as to cause
or accelerate said decomposition of said L-aspartic acid
20 derivative in the absence of said stabilizer. Also, the
present invention relates to a process for preparing and
storing a comestible composition that is sweetened at least
partially with at least one L-aspartic acid sweetening agent
derivative freely dispersed, as such, therein, and wherein
25 said composition has a moisture content and/or a heat history
which, unless stabilized against, upon the normal commercial
storage of said composition would cause or accelerate the
decomposition of said L-aspartic acid derivative into
decomposition products which are devoid of sweetening
30 characteristics, the improvement which comprises formulating
said composition with, as a stabilizer for said L-aspartic
acid derivative and freely dispersed in said composition, on

1 a wet basis, about 10 to 30% of cooked hydrogenated starch
hydrolysate, and storing said composition and recovering at
least 70% of the formulated amount of said L-aspartic acid
derivative after said storage for at least twelve months.

5 According to the present invention, chewing gum
products containing $\geq 2\%$ moisture and sweetened, at least in
part, with aspartame, can have such aspartame stabilized to a
relatively high degree against decomposition into
decomposition products such as diketopiperazine, by
10 formulating the product with stabilizing amounts of aqueous
hydrogenated starch hydrolysate having a cooked moisture
content of about $10 \pm 6\%$.

The cooked aqueous hydrogenated starch hydrolysate
used in the compositions of the present invention as a
15 stabilizer for the aspartame has a moisture content of about
 $10 \pm 6\%$, and preferably of about $10 \pm 4\%$. This cooked
hydrogenated starch hydrolysate is prepared by cooking, or
heating, commercially available aqueous solutions of the
hydrogenated starch hydrolysate, which have moisture contents
20 of about 20 to 35%, at a temperature of about 250 to 310°F,
and preferably of about 260 to 290°F, until the desired low
moisture content of the cooked material is attained. This
may take about 0.5 to 1.0 hours for a 100 gallon batch of a
commercially available aqueous solution of hydrogenated
25 starch hydrolysate having a moisture content of about 25%.

The uncooked commercially available hydrogenated
starch hydrolysate which may be used to form the cooked
hydrogenated starch hydrolysate used in the compositions of
the present invention may be a hydrogenated corn syrup or
30 hydrogenated starch hydrolysate of varying dextrose
equivalents (DE), such as are disclosed in U.S. Patent Re.
No. 26,959 and U.S. Patent Nos. 3,556,811, 4,279,931 and
4,382,962, as well as various hydrogenated glucose syrups

1 and/or reconstituted powders which contain sorbitol,
hydrogenated disaccharides, tri- to hexa-hydrogenated
saccharides, and hydrogenated higher polysaccharides, or
mixtures of any two or more of the above.

5 The uncooked commercially available hydrogenated
glucose syrups or hydrogenated starch hydrolysates and/or
powders thereof may be produced by catalytic hydrogenation of
standard glucose syrups (acid and/or enzyme converted) to the
point where all the glucose end groups of the saccharides are
10 reduced to alcohols, that is, dextrose to sorbitol. In the
case of hydrogenated glucose syrups, the total solids
contents will usually range from about 65 to about 80%, which
solids are made of from about 4 to 70%, and preferably from
about 4 to about 20%, sorbitol, from about 8 to about 65%,
15 and preferably from about 20 to about 65%, hydrogenated
disaccharides (that is, mannitol), and 20 to 80% of the
higher (\geq tri to hepta) hydrogenated saccharides. The
preferred of the uncooked commercially available hydrogenated
starch hydrolysates contain from about 8 to about 45%, and
20 preferably about 15 to 45%, tri- to hepta-hydrogenated
saccharides, and from about 10 to about 35%, and preferably
about 15 to 25%, hydrogenated saccharides higher than hepta.

The commercially available hydrogenated starch
hydrolysate is also referred to in the literature as
25 hydrogenated glucose syrup, or by the trademark or tradename
Lycasin polyol or Lonza polyol. The term hydrogenated starch
hydrolysate will be used herein to designate such material.
The hydrogenated starch hydrolysate is usually sold
commercially in the form of an aqueous solution thereof
30 having a moisture content of about 20 to 35%.

The chewing gum compositions of the present
invention are made with about $20 \pm 10\%$, and preferably, about
 $15 \pm 5\%$, of the cooked hydrogenated starch hydrolysate. When
less than about 10% of the cooked hydrogenated starch

1. hydrolysate is used the resulting products have poor flexibility properties, and when more than about 30% of the cooked hydrogenated starch hydrolysate is used, the resulting product is too soft.

5 The chewing gum products of the present invention, have a moisture content of about 2 to 8%, and preferably of about 2 to 5%

10 The chewing gum products of the present invention may also comprise about ≥ 0 to 12%, and preferably about 5 to 9%, glycerine.

15 The glycerine assists in providing long term storage flexibility, and initial softness of chew. The glycerine may be cooked with the hydrogenated starch hydrolysate and added, as such, to the chewing gum formulation, or it may be added separately. When the glycerine is cooked with the hydrogenated starch hydrolysate, the cooking temperature should not exceed the decomposition temperature of the glycerine, which is about 290°C.

20 The chewing gum products of the present invention have ERH values of about > 25 to 50, and preferably of about 30 to 50, and most preferably about 30 to 40.

25 The cooked hydrogenated starch hydrolysate and/or the glycerine may be added to the gum base portion, or with the non-gum base components, when formulating the products of the present invention. The cooked hydrogenated starch hydrolysate, with or without glycerine cooked therewith, is cooled to below 150°F before being admixed with the other components of the chewing gum products of the present invention.

30 When the cooked hydrogenated starch hydrolysate is prepared (cooked) with less than about 4% glycerine, the resulting product is in a glossy solid state at room temperature. When the cooked hydrogenated starch hydrolysate is made with about 4 to 12% glycerine, the resulting product
35 is liquid or is pourable at room temperature.

1 This solid glossy material melts, or becomes
pourable, at a temperature of about 212 to 220°F. When using
cooked hydrogenated starch hydrolysate in the chewing gum
products of the present invention which has been made with
5 less than about 4% glycerine, it is preferable, to facilitate
the incorporation of the cooked material into the chewing gum
composition, to use the cooked hydrogenated starch
hydrolysate just after it has been cooked, and it is still in
a heated and liquid state. It is preferable under such
10 circumstances to allow the cooked hydrogenated starch
hydrolysate (made with less than 4% glycerine) to cool down
to about 150 to 160°F before incorporating it into the
chewing gum formulation.

 When the cooked hydrogenated starch hydrolysate is
15 made with about 4 to 12% glycerin, it is preferable to cool
it down to about 100 to 110°F before it is added to the
chewing gum formulation.

 The preferred L-aspartic acid derivative to be used
in the compositions of the present invention is, as noted
20 above, L-aspartyl-L-phenylalanine methyl ester, known as
aspartame. Other L-aspartic acid sweetening derivatives may
also be used. Such derivatives are disclosed in U.S. Patent
No. 3,955,000 at column 3, line 63 to column 4, line 35, the
disclosure of which is incorporated herein by reference. The
25 following description will be directed to aspartame with the
understanding that the other L-aspartic acid sweetening
derivatives may also be used in lieu of and/or in addition to
the aspartame. These compounds are also known as dipeptides.

 The compositions of the present invention contain
30 about 0.01 to 2.0, and preferably about 0.1 to 0.25%, of the
aspartame.

 The chewing gum products of the present invention
do not contain a liquid filling, they are completely solid
materials.

1 When made in stick form, the chewing gum products
of the present invention are flexible. When prepared in
stick form the sticks are about 0.065 to 0.072 inch in
thickness, about 3/4 inch in width and about 2 7/8 to 3
5 inches in length.

The flexibility of the stick products of the
present invention and those of the prior art products can be
evaluated by the following test procedure:

A single stick of gum, which is at room
10 temperature, i.e., 20-25°C, is held between the thumb and
index finger of both hands, along the wide sides of the
stick, and within 0.5 to 1.0 inch of each end of the stick,
and is then slowly twisted in opposite directions relative to
the long axis of the wide side of the stick. A stick of gum
15 made according to the present invention, which has good
flexible properties, will bend or shape, without breaking or
crumbling, after at least two or three 180° turns of the
fingers, into a helical shaped structure before tearing.

A non-flexible stick of gum, on the other hand will
20 typically break up or crumble after, or before, the
completion, of only one 180° turn of the fingers.

The flexibility properties of stick chewing gum
products made according to the present invention will last
during prolonged storage of such products, i.e., for at least
25 9 to 12 months, when the products are stored at 40 to 70%
relative humidity and at 20 to 30°C.

Although many prior art sugarless stick regular
chewing gum products may pass the above described flexibility
test when they are freshly made, and are warm, they will not
30 do so after they have cooled down to room temperature i.e.,
20-25°C.

CHEWING GUM COMPOSITIONS

The chewing gum compositions contemplated by the
present invention comprise all types of sugarless chewing

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1 gums and chewing gum formulations known to those skilled in
the art, including the regular gum and the bubble gum types.
Typical chewing gum compositions comprise a chewing gum base,
5 other additives such as flavoring agents, colorants and
antioxidants. The modifying agents are used to soften,
plasticize and/or compatibilize one or more of the components
of the gum base and/or of the formulation as a whole.

10 The chewing gum products of the present invention
would have the following formulation:

		Weight Percent of Component	
		<u>Broad Range</u>	<u>Preferred Range</u>
	Component		
	gum base	15 to 35	20 to 30
	cooked hydrogenated		
	starch hydrolysate	10 to 30	10 to 20
15	glycerin	0 to 12	5 to 9
	modifying agent other		
	than glycerin	0 to 5	0.3 to 3.0
	aspartame	0.01 to 2	0.1 to 0.25
20	non-nutritive sweetener	0 to 2	0.1 to 0.4
	(other than aspartame)		
	bulking agent or bulk		
	sweetener	20 to 70	30 to 50
	coloring agent	0.1 to 0.5	0.15 to 0.3
	flavoring agent		
25	(other than sweetener)	0.5 to 2.5	0.18 to 1.2
	moisture*	<u>2 to 8</u>	<u>2.0 to 5.0</u>
	Total	100	100

30 * Moisture content contributed by all components.

GUM BASE

1 The composition of the gum base will vary depending
 on whether the gum base is to be used in a chewing gum
 product which is to be a regular, or non-bubble, gum product
 5 or a bubble gum product. For use in making a bubble gum or
 regular chewing gum product, the following gum base
 formulations may be used, in accordance with the present
 invention:

	Component	Weight percent of component in Gum base for			
		<u>Bubble Gum Product</u>		<u>Regular Gum Product</u>	
		<u>Broad</u> <u>Range</u>	<u>Preferred</u> <u>Range</u>	<u>Broad</u> <u>Range</u>	<u>Preferred</u> <u>Range</u>
10	Masticatory				
15	material	8-22	9-18	8-25	9-18
	plasticizer				
	for masti-				
	catory				
20	material	5-35	10-20	2-30	8-20
	hydrophilic				
	detackifier	0-30	4-10	5-35	10-25
25	plasticizer				
	for hydro-				
	philic				
	detackifier	0-14	0-8	1-15	3-12
30	wax	3-15	5-10	4-20	8-15
	mineral				
	filler	1-35	10-22	5-35	15-30
35	antioxidant	0-0.1	0.05-0.09	0-0.1	0.03-0.09
	Total		100		100

1 The masticatory substances are elastomeric
materials which may be synthetic or natural in origin. The
masticatory substances of synthetic origin would include
styrene-butadiene copolymer, butyl rubber (which is
5 isobutlyene-isoprene copolymer) and polyisobutlyene. The
natural masticatory substances would include chicle, crown
gum, nispero, balata, jelutong, pendare, perillo, niger
gutta, tunu, leche caspi, sorva and gutta hank kang.

 The plasticizer for the masticatory substances will
10 preferably comprise a hydrogenated ester gum, that is, a
glycerol ester of hydrogenated rosin and/or dimerized ester
gum. However, other resins may be employed such as
pentaerythritol ester gum, polymerized ester gum, polyterpene
resin and ester gum.

15 The hydrophilic-type detackifier is a material
which will absorb saliva and would include vinyl polymers
having a molecular weight of at least 2,000, and preferably
of about 2,000 to 80,000 or more, such as polyvinyl acetate,
polyvinyl butyl ether and copolymers of vinyl esters and/or
20 vinyl ethers with ethylene.

 The plasticizers for the hydrophilic type
detackifiers would include one or more of triacetin,
acetylated glycerides and other flavor adjuvants such as
ethyl acetate and triethyl citrate, and others as listed in
25 U.S. Patent No. 4,452,820 at column 4, lines 27 to 46, the
disclosure of which is incorporated herein by reference.

 The oleaginous material includes waxes which are
used primarily as compatibilizers/plasticizers between the
elastomeric and resin phases, where such two phases are
30 employed. Examples of the waxes are petroleum waxes such as
paraffin wax and microcrystalline wax; the polyethylene
waxes; and natural waxes derived from either plant or animal

1 sources such as candelilla wax, carnuba wax and bees wax.
The oleaginous material may also include hydrogenated
vegetable or animal fats, cocoa butter or other softening-
emulsifying agents such as phosphatides such as lecithin and
5 di- and tri-glycerides of fatty acids.

The mineral fillers would include calcium carbonate,
titanium dioxide, talc, alumina, tricalcium phosphate and
mixtures thereof. The compositions of the present invention
can be formulated with materials such as calcium carbonate
10 without having a deleterious effect on the stability of the
aspartame.

In addition, the gum base may include antioxidants
such as butylated hydroxy toluene, butylated hydroxy anisole
and propyl gallate.

15 If desired, the aspartame may be employed together
with another non-nutritive or artificial or intense sweetener
such as poorly water-soluble, as well as water-soluble
sweeteners such as the free acid form of saccharin, sodium,
calcium or ammonium saccharin salts, dihydrochalcones,
20 glycyrrhizin, dipotassium glycyrrhizin, glycyrrhizic
acid/ammonium salt, talin, acesulfame K, as well as Stevia
rebaudiana (Stevioside), Richardella dulcifica (Miracle
Berry), Dioscoreophyllum cumminisum (Serendipity Berry), free
cyclamic acid and cyclamate salts, and the like, or mixtures
25 of any two or more of the above.

The artificial sweetener may be employed with
non-sugar bulk sweeteners (other than the cooked hydrogenated
starch hydrolysate) such as dry crystalline sugar alcohols
such as sorbitol, xylitol and mannitol. The preferred
30 products of the present invention are made without sugar or
other nutritive sweeteners.

In addition to the above, the chewing gum made by
this invention can also contain conventional FD&C and natural
coloring agents.

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- 1 The flavoring which can be included in the chewing
gum compositions made according to this invention can
comprise one or more natural and/or synthetic flavors and/or
oils derived from plants, leaves, flowers and fruit.
- 5 Representative flavors and oils of these types include acids
such as adipic, succinic and fumaric acid; citrus oils such
as lemon oil, orange oil, lime oil and grapefruit oil; fruit
essences, such as apple essence, pear essence, peach essence,
strawberry essence, apricot essence, raspberry essence,
10 cherry essence, plum essence and pineapple essence; essential
oils such as peppermint oil, spearmint oil, mixtures of
peppermint oil and spearmint oil, clove oil, bay oil, anise
oil, oil of nutmeg, oil of sage, oil of bitter almonds,
cassia oil and methylsalicylate (oil of wintergreen).
- 15 Various synthetic flavors, such as those for a mixed fruit,
may also be incorporated in the chewing gum with or without
conventional preservatives.

GENERAL PREPARATION OF CHEWING GUM PRODUCT

- 20 The chewing gum products of the present invention
are prepared by first separately preparing the gum base. To
then prepare the sugarless chewing gum formulation, the gum
base for the product is melted, at a temperature about 190
to 250°F, and the other components of the composition are
added thereto. The resulting composition is uniformly
25 admixed. Each of the components is usually separately added
to the formulated composition and uniformly mixed in before
the next component is added. All of the admixing operations
are conducted at temperatures in the range of about 115 to
185°C, and preferably of about 115 to 130°C, for a total
30 mixing time, at such temperatures, of about 10 to 20 minutes.
These operations do not have to be conducted under anhydrous
conditions in preparing the compositions of the present

1 invention, and any amounts of moisture that are normally
present in the raw materials, other than the uncooked
hydrogenated starch hydrolysate, that are used in the
compositions of the present invention do not usually have to
5 be removed therefrom either prior to, or during, the
formulating process. The one exception to this concept of
not removing water occurs when using rubber latices as the
source of the masticatory substance. As in prior art
practice, the moisture content of the latex is, essentially,
10 normally removed after coagulating the latex.

The chewing gum formulations disclosed herein may
thus be prepared, and processed into chewing gum products,
using conventional chewing gum formulation mixing, processing
and packaging equipment and concepts.

15 The following examples are merely illustrative of
the present invention and are not intended as a limitation
upon the scope thereof.

PREPARATION OF CHEWING GUM COMPOSITIONS

Various chewing gum compositions were prepared
20 using various of the gum bases prepared as described above.
In preparing the chewing gum compositions, they were prepared
in pilot plant sized batches in paddle mixers. In preparing
each batch, the previously prepared gum base is melted at a
temperature of up to about $245^{\circ} \pm 5^{\circ}\text{F}$ and is premixed with
25 lecithin and color additive, and, optionally, the cooked
hydrogenated starch hydrolysate, and glycerine, if the latter
is used, until the temperature drops to about 185 to 189°F .
Then there is sequentially added powdered sorbitol (at a
temperature of $\leq 180^{\circ}\text{F}$), liquid flavor (i.e., peppermint),
30 liquid sorbitol, if used, glycerin, if used, alone or with
the cooked hydrogenated starch hydrolysate, if the latter is
not premixed with the gum base, any additional flavorants,

- 1 and finally the aspartame, and, optionally, any other intense
sweeteners. Each component is paddle mixed in before the
next is added. Each mixing step takes about 0.5 to 5.0
minutes and the total mixing time is about 10-20 minutes.
5 The resulting product is recovered and further processed and
packaged using conventional chewing gum making procedures.

The cooked hydrogenated starch hydrolysate used in
the chewing gum compositions evaluated in the Examples had a
moisture content of 7.5% or 11.5% and was prepared by
10 cooking, at 250 to 310°F, a commercially available aqueous
hydrogenated starch hydrolysate which had a solids content of
75 ± 1%, a monosaccharide content of about 6 to 8%, a
disaccharide content of about 50 to 55%, and a higher
saccharide content of about 38 to 48%. The higher
15 saccharides were about 20 to 25% in the 3-7 DP (degree of
polymerization) range, and about 18 to 23% in the > 7 DP
range.

Unless otherwise indicated, the total water content
reported below for each of the chewing gum formulations of
20 the Examples is a calculated amount based on the water
content of the cooked hydrogenated starch hydrolysate (HSH),
about 10 ± 6%, and glycerine, about 1 to 4%, (where used)
plus any added water used in the respective formulations.
The actual water content of these formulations is about 0.2
25 to 0.5% higher than the reported calculated values, since
such additional amounts of water enter the final formulated
product from the other components of the formulation and from
the ambient atmosphere. The total of the reported weight
percentages for the respective formulations of these Examples
30 will thus total about 100% plus the respectively reported
calculated water contents.

1 The test formulations were prepared using batch
pilot plant procedures. Prior to beginning the pilot plant
batch making process, a batch of cooked HSH/glycerine was
prepared, and then split into two half batches each of which
5 contains one-half of each of the formulated amounts of the
glycerine and the cooked hydrogenated starch hydrolysate that
are to be used in making the product of each example. One of
such premixtures is then used in each of steps 3 and 6 noted
below.

10 STEP-WISE PILOT PLANT PROCEDURE

1. Into a pre-warmed sigma bladed mixer add molten
gum base. The gum base temperature should be between
150-200°F, and preferably between 170-190°F.

2. With the blades of the mixer operating, and the
15 temperature in the cited range, the lecithin is added and the
mixing is continued for one minute.

3. Add one half of the cooked HSH/glycerine
premixture, or the first 1/2 of the HSH, and mix for two
minutes, or until homogeneous.

20 4. Add 1/2 of the sorbitol powder and continue
mixing for two minutes.

5. The liquid flavor is then added and mixing is
continued for one minute.

6. Add the second half of the cooked HSH/glycerine
25 premixture, or the second 1/2 of the cooked HSH, and mix for
two minutes.

7. Add the remaining sorbitol powder and the
non-nutritive sweetener. Prior to production of the batch,
the non-nutritive sweetener is premixed with a small portion
30 of the sorbitol powder. Continue mixing for two minutes, or
until the batch is homogeneous. The final gum temperature is
approximately 112°F.

1 8. The gum is removed from the mixer and
conditioned at 70°F/42-55% R.H. prior to forming.

5 9. The gum is rolled and scored into a stick
configuration. Mannitol is applied to the surface of the
scored gum to prevent surface adhesion. The gum is then
packaged in fin-seal foil pouches and sealed.

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Examples 1 and 2

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Using the pilot plant procedure described above two (2) chewing gum products were prepared from the following formulations, in % by weight.

5	Component	Weight % of Compound		
		Example	1	2
	styrene-butadiene		30	30
	copolymer based gum base			
10	sorbitol, powder		43	40
	cooked hydrogenated starch hydrolysate		20	20
15	glycerine		5	8
	flavor		1	1
20	lecithin		1	1
	aspartame		0.2	0.2
	moisture	Ca.	3	Ca. 2.2
25			100	100
	Ca. = about			

The cooked hydrogenated starch hydrolysate used in Example 1 and 2 had a moisture content of, respectively, 11.5 and 7.5%.

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1 The chewing gum products of the present invention
were flexible, in stick form. When tested for flexibility,
as described above, individual sticks of gum made from each
of the Example 1 and 2 products were twisted 3 times before
5 breaking. These tests for flexibility were conducted,
periodically, over a storage period of up to about 55 days
under accelerated aging conditions of 100-105°F and about 30%
relative humidity.

10 The packaged chewing gum products of the present
invention made in Examples 1 and 2 were also evaluated for
the shelf life stability of the aspartame used therein while
stored for up to about 40 to 55 days under accelerated aging
conditions at 100-105°F and ambient, about 30°, relative
humidity with the following results:

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TABLE 1

% APM Recovery During
Accelerated Aging Storage of
Example 1 and 2 Chewing Gum
Samples

1								
5	Days Storage	0	14	21	28	35	41	56
	% APM							
10	Recovery							
	Example 1	93	NT	86.5	-	-	79.5	-
	Example 2	96	94.5	NT	81.5	80	-	74

NT = not tested at this time interval

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1 The APM was analyzed for, employing standard gas
chromotography procedures, first at zero time, i.e., within
48 hours after the product was first made and prior to its
being subjected to any accelerated aging, and then at various
5 intervals, as noted in Table I above, after 14 to 56 days of
accelerated aging time. Based on experience, each day of
accelerated aging time is equivalent to about 7 to 7.5 days
of commercial storage aging time. Thus 40 days of
10 accelerated aging time is equivalent to about 280 to 300
days, or about 10 months, of commercial storage time, and 50
days of accelerated aging time is equivalent to about 350 to
375 days, or at least one year, of commercial storage time.
These test results thus indicate that the aspartame is very
stable in the compositions of the present invention in that
15 at least 80% of the APM is retained and is recoverable for at
least 40 days of accelerated aging and at least 70% of the
aspartame is retained and is recoverable for at least 50 days
of accelerated aging time.

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Examples 3 to 8

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A series of six sugarless stick chewing gum products were evaluated for ERH values and flexibility values. The compositions of these products are indicated below, or in Table 2.

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Products 3 and 4 were made according to the present invention, as described above, using either a styrene-butadiene based gum base (Gum Base A) or a gum base made from an admixture of styrene-butadiene elastomer and polyisobutylene elastomer (Gum Base B). Products 5 and 6 were made with Gum Base A and were based on prior art composition technology (high glycerine, low moisture content). Products 5 and 6 were otherwise prepared as described above. Product 7 is an off-the-shelf sample of Trident spearmint gum manufactured by Warner Lambert Co. Product 8 was an off-the-shelf sample of Extra bubble gum manufactured by Wm. Wrigley Jr. Company. (Trident and Extra are trademarks of such companies).

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The list of contents of the Extra product, as indicated on the label thereof, is sorbitol, gum base, mannitol, glycerol, artificial and natural flavors, lecithin, aspartame, artificial colors and butylated hydroxy toluene.

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The list of contents of the Trident product, as indicated on the label thereof, is sorbitol, gum base, glycerine, mannitol, softeners, natural and artificial flavors, and sodium saccharin.

25

The compositions of the Trident and Extra products thus do not contain hydrogenated starch hydrolysate in any form.

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It is believed that the Trident product contains about 2% water and that the Extra product contains about 1% water. The exact formulated contents of these products is not known.

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1 The products were each analyzed for their ERH
value, as described above. The ERH values of these products
are shown below in Table 2.

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TABLE 2
Example 3-8 Formulations

Component	3	4	5	6	7	8
5 Gum base	27.0	27.0	30.0	30.0	-	-
sorbitol powder	47.4	46.3	60.0	50.0	-	-
cooked HSH	15.0	16.5	0	0	-	-
glycerine	8.0	8.0	8.0	18.0	-	-
10 peppermint oil	1.2	1.2	1.0	1.0	-	-
lecithin	1.2	0.7	1.0	1.0	-	-
aspartame	0.2	0.2	0.2	0.2	-	-
colorant	0	0.1	0	0	-	-
% water	2 3	2 3	1 2	1 2	ca. 2	ca.1
15 ERH Value:	37	35	14.6	14.0	25	19

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1 When tested for flexibility, as described above,
only Products 3 and 4 were flexible. Thus, only Products 3
and 4 had the desired combination of flexibility and high ERH
values.

5 In addition to being useful in chewing gum
products, aspartame stabilized with the cooked hydrogenated
starch hydrolysate can also be used in other comestibles or
ingested products which might employ a bulk non-nutritive
sweetener such as toothpaste, confectionary products
10 including the chewing gum products, including the regular
non-bubble gum as well as the bubble gum types, hard candy,
liquid filled chewing gum and candy, medicinals such as cough
drops, antacids, and breath fresheners; bakery goods such as
cookies and others such as salad dressings and puddings.

15 The aspartame need not be encapsulated or coated in
any way when employed in the products of the present
invention. It and the other components of such products in
which it is used may all be freely dispersed in such
products.

20 The use of cooked hydrogenated starch hydrolysate,
as a stabilizer for the L-aspartic acid derivatives in the
moisture containing comestible products of the present
invention enables the formulator to use about 30 to 50% less
of the L-aspartic acid derivative, and still recover th same
25 amounts of L-aspartic acid derivative over the extended
storage periods noted above, as when the L-aspartic acid
derivative is used in the same comestible product but without
the cooked hydrogenated starch hydrolysate stabilizer of the
present invention.

30 The use of the cooked hydrogenated starch
hydrolysate stabilizers of the present invention is also
useful in stabilizing comestibles such as the chewing gum

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1 compositions prepared above which have manufacturing heat
histories of at least 115°C for at least 10-20 minutes and
which heat histories may also have a deleterious effect on
the stability of the L-aspartic acid sweeteners used wherein.

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WHAT IS CLAIMED IS:

1. A comestible composition sweetened at least partially with at least one L-aspartic acid sweetening agent derivative freely dispersed therein and comprising stabilizing amounts of a stabilizer adapted, upon the normal commercial storage of said composition for at least twelve months, to retard or prevent the decomposition of said L-aspartic acid derivative into decomposition products which are devoid of sweetening characteristics to such extent as to allow for the recovery of at least 70% of the formulated amount of said L-aspartic acid derivative after said storage, said stabilizer comprising cooked hydrogenated starch hydrolysate, and said composition comprising moisture in such amounts as to cause or accelerate said decomposition of said L-aspartic acid derivative in the absence of said stabilizer.
2. A composition according to Claim 1 wherein the composition contains $\geq 2\%$ moisture and the cooked aqueous stabilizer has a moisture content of $10\% \pm 6\%$.
3. A composition according to Claim 1 or 2 wherein the cooked stabilizer has a moisture content of $10 \pm 4\%$ and the composition has a moisture content of 2 to 8%.
4. A composition according to any of Claims 1 to 3 wherein the compositions contain up to 12% glycerene.
5. A composition according to any of Claims 2 to 4 wherein the formulated amount of the L-aspartic sweetening agent derivative is about 30 to 50% less than the amount of said L-aspartic acid sweetening agent that would have had to have been formulated into said composition, in the absence of the starch hydrolysate stabilizer, to provide the recoverable amount of the L-aspartic acid sweetening agent under the storage conditions, the moisture, L-aspartic acid sweetening agent and the stabilizer each being freely admixed into said composition, and said percents being weight percents.

- 1 6. A composition according to Claim 1 comprising,
in weight percent,
 i) about 2 to 8% moisture
 ii) about 0.01 to 2.0% L-aspartic acid
5 sweetening agent derivative, and
 iii) as a stabilizer for said L-aspartic acid
derivative, stabilizingly effective quantities of cooked
hydrogenated starch hydrolysate having a moisture content of
about 10 ± 6%,
 said composition being adapted, upon the storage
10 thereof at 30° relative humidity and 100-105°F, to retain at
least 80% of said L-aspartic acid derivative therein for at
least 40 days.
7. A composition according to any of Claims 1 to 6
15 in which the L-aspartic acid derivative is aspartame.
8. A composition according to Claim 6 wherein 0.10
to 0.25% aspartame is present.
9. A composition according to any of Claims 1 to 8
further comprising the following additional components,
- 20 15 to 35% gum base
 0 to 5.0% gum base modifier
 20 to 70% bulk sweetening agent
 0.1 to 0.5% coloring agent
 0.5 to 2.5% flavoring agent
25
10. A composition according to any of Claims 1 to 9
having a cooked hydrogenated starch hydrolysate content of
about 10 to 30 weight %.
- 30 11. A composition according to any of Claims 1 to
10 in the form of a flexible stick chewing gum product.
12. A composition according to Claim 11 wherein the
chewing gum product has an ERH value of >25 to 50.
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13. A process for preparing and storing a
1 comestible composition that is sweetened at least partially
with at least one L-aspartic acid sweetening agent derivative
freely dispersed, as such, therein, and wherein said
5 composition has a moisture content and/or a heat history
which, unless stabilized against, upon the normal commercial
storage of said composition would cause or accelerate the
decomposition of said L-aspartic acid derivative into
decomposition products which are devoid of sweetening
10 characteristics, the improvement which comprises formulating
said composition with, as a stabilizer for said L-aspartic
acid derivative and freely dispersed in said composition, on
a wet basis, about 10 to 30% of cooked hydrogenated starch
hydrolysate, and storing said composition and recovering at
15 least 70% of the formulated amount of said L-aspartic acid
derivative after said storage for at least twelve months.

14. A process according to Claim 13 wherein the
composition has a moisture content of $\geq 2\%$ and contains 0.01
to 2% aspartame, and the stabilizing of said aspartame
20 against the destabilizing effects of said moisture with such
stabilizingly effective amounts of cooked hydrogenated starch
hydrolysate having a moisture content of about $10 \pm 6\%$ as to
provide for the recovery of at least 70% of said formulated
amount of aspartame after said comestible product has been
25 subject to ambient commercial storage conditions for at least
twelve months.

15. A process as in Claim 14 in which said
aspartame is stabilized so as to allow for the recovery of at
least 80% of said amount of aspartame after at least ten
30 months of ambient commercial storage.

16. The use of cooked aqueous hydrogenated having a
moisture content of $10 \pm 6\%$ in the preparation of a
comestible containing aspartame.

17. A use as in Claim 16 wherein said comestible is
35 chewing gum.



European Patent
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EUROPEAN SEARCH REPORT

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Application number

EP 86 10 4310

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.4)
X	WO-A-8 501 862 (GENERAL FOODS CORP.) * page 7, lines 10-12, examples 3, 4 *	1, 4, 11	A 23 L 1/236 A 23 G 3/30
D,A	US-A-4 248 895 (J.J. STROZ et al.) * example 5, claims 1, 4, 6 *		
D,A	US-A-4 384 004 (T. CEA et al.) * abstract, claims 1, 10 *		
A	EP-A-0 097 950 (AJINOMOTO CO.) * claim 1 *		
			TECHNICAL FIELDS SEARCHED (Int. Cl.4)
			A 23 L 1/00 A 23 G 3/00
The present search report has been drawn up for all claims			
Place of search BERLIN		Date of completion of the search 20-06-1986	Examiner SCHULTZE D
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